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(54) Laundry detergent tablets

(57) Laundry detergent tablets which in addition to usual adjuvants and additives comprise 0.1 to 15% by weight of nonionic tenside, 0.1 to 15% by weight of amphoteric tenside, 20 to 50% by weight of a polyfunctional carboxylic acid and/or a salt thereof, calculated as trisodium citrate dihydrate, 1 to 30% by weight of layered silicate and/or alkalimetal silicate and/or zeolite as well as 5 to 70% by weight of potassium carbonate. Further ingredients advantageously included in the laundry de-

tergent tablets are sodium carbonate and/or potassium carbonate, sodium bicarbonate and/or potassium carbonate, disintegrating agents, binders, enzymes, anti-foaming agents, agents preventing the running of colour, antiredepositing agents, polymers and complex-binding agents. The laundry detergent tablets have a good washing effect at the same time as they have a good storage stability and dissolve quickly in the washing water.

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DescriptionTechnical Field

5 The present invention relates to laundry detergent tablets which are advantageous in having a good washing effect, and which are simultaneously very good storage stability and easily soluble in the washing water.

Background Art

10 Conventionally used laundry detergent compositions can be divided into two main types, viz. liquid and powder washing compositions.

During recent years laundry detergent tablets have, however, been increasingly focused on because they present the advantage of being easy to handle and to dose correctly.

15 However, many requirements are presented to laundry detergent tablets, viz. both technical requirements and requirements to the contents thereof. These requirements often counteract one another, and accordingly it is very difficult to formulate laundry detergent tablets which are satisfactory in all aspects.

Thus the laundry detergent tablets must ideally meet the following requirements:

- have sufficient tablet strength so that they do not crumble or break while stored, during transport or while handled.
- 20 - have a good storage stability and be able to withstand storage under various climatic conditions involving fluctuating temperatures and humidities,
- be easily soluble in the washing water in such a manner that they can exert their effect.
- 25 - have a good washing effect, which requires a high content of active washing substances, and be able to exert this washing effect to an optimum across a wide spectrum of washing programmes at different temperatures and water hardness degrees.

30 A sufficient tablet strength can be obtained by increasing the pressure used for compressing the laundry detergent tablets. An increase of the pressure has, however, a negative effect on the solubility of the laundry detergent tablets, and accordingly these properties are very difficult to combine.

The demand for a quick dissolving of the laundry detergent tablets in the washing water is further sharpened by the growing tendency of performing the machine-wash using a decreasing amount of water, lower temperatures and shorter washing periods for environmental and financial reasons and in order to obtain consumer acceptance.

35 The demand for a good washing effect involves as mentioned above a requirement for a high content of active washing substances having a tendency to extend the disintegration period of the cloth detergent tablets and to reduce their shelf life.

JP No. 6279799 (LION CORP.) describes detergent tablets based on 1 to 50% by weight of an anionic tenside in form of an alpha--sulpho-fatty acid derivative as well as 5 to 60% by weight of an alkali carbonate, said tablets being described as being easily soluble in water. The alkali carbonate is potassium carbonate or a mixture of potassium carbonate and sodium carbonate in a weight ratio of from 100:0 to 40:60 and has preferably an average particle size of less than 300 µm, especially less than 150 µm.

45 JP No. 6108099 (LION CORP.) describes easily soluble detergent tablets with a high washing effect and containing 5 to 50% by weight of one or more surfactants, including nonionic surfactants, 10 to 70% by weight of potassium carbonate as well as 0.5 to 5% by weight of pulverulent perfume.

JP No. 6017099 (LION CORP.) describes easily soluble detergent tablets with a high washing effect and containing (A) 1 to 50% by weight of an anionic surfactant, (B) 0.1 to 4% by weight of a nonionic surfactant, as well as (C) potassium carbonate in a weight ratio of (B):(A) of up to 0.5 and a weight ratio of (B):(C) of up to 0.25.

50 JP No. 4239100 (LION CORP.) describes easily soluble detergent tablets containing 5 to 50% by weight of one or more surfactants, including nonionic surfactants, as well as 10 to 70% by weight of potassium carbonate. The potassium carbonate used has preferably an average particle size of up to 150 µm.

None of these LION-publications describe, however, detergent tablets based on a tenside system of a nonionic tenside and an amphoteric tenside as well as a zeolite-poor or -free builder system of a polyfunctional carboxylic acid and/or a salt thereof, such as citrate, and layered silicate (in German known as "Schicht-silikat") and/or alkali metal silicate and/or zeolite.

55 EP No. 0 482 627 A1 (Kao Corporation) discloses laundry detergent tablets containing potassium carbonate and a nonionic surfactant with an HLB value of from 8.0 to 16.0 in a weight ratio of from 5:1 to 1:5.

All the recipes indicated in the publication contain relatively high amounts of zeolite and are based on a nonionic surfactant as well as optionally an anionic surfactant unlike the present laundry detergent tablets. The present laundry detergent tablets are based on a combination of a nonionic tenside and an amphoteric tenside as well as a zeolite-poor or -free builder system of a polyfunctional carboxylic acid and/or a salt thereof, such as citrate, and layered silicate and/or alkali metal silicate and/or zeolite.

These known laundry detergent tablets are indicated to be easily soluble in the washing water.

As it appears from the following comparison test, it turned out to be very difficult to prepare these tablets in a conventional manner by means of a tableting machine. In Example 1 and Example 2 in the publication, the preparation of tablets is described by way of melt kneading followed by a moulding in pressurized cylinders, and in Example 1 the pressurized moulding is indicated to last 1 minute where the production speed at conventional tableting by way of comparison is from 25,000 to 90,000 tablets per hour. Such a moulding process is therefore highly disadvantageous and uneconomic for the preparation of tablets on an industrial scale.

The comparison test revealed that the laundry detergent tablets according to the invention showed a disintegration period being far superior to the disintegration period of the laundry detergent tablets described in EP No. 0 482 627 A1.

Thus the object of the present invention is to provide easily soluble laundry detergent tablets having a high washing effect and a good storage stability.

Brief Description of the Invention

The above object is surprisingly obtained by the laundry detergent tablets according to the invention, which are characterized in that in addition to the optional, conventional adjuvants and additives they comprise:

nonionic tenside	0.1 to 15% by weight
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amphoteric tenside	0.1 to 15% by weight
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polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
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layered silicate and/or alkali metal silicate and/or zeolite	1 to 30% by weight
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potassium carbonate	5 to 70% by weight
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According to a preferred embodiment of the invention, the laundry detergent tablets comprises furthermore one or more ingredients selected from sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate, disintegrating agents, binders, enzymes, antifoaming agents, agents preventing the running of colour, antiredepositing agents, such as cellulose colloids, such as carboxymethyl cellulose, polymers and complex-binding agents, such as phosphonates.

Thus according to a preferred embodiment the laundry detergent tablets according to the invention comprise in addition to optional, usual adjuvants and additives

	nonionic tenside	0.1 to 15% by weight
5	amphoteric tenside	0.1 to 15% by weight
10	polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
15	layered silicate and/or alkali metal silicate, and/or zeolite	1 to 30% by weight
	potassium carbonate	5 to 70% by weight
20	disintegrating agent	0.5 to 5% by weight
	binder	1 to 8% by weight
25	sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate	0 to 10% by weight
30	enzyme	0 to 10% by weight
	antifoaming agent	0 to 1% by weight
35	agent preventing the running of colour	0 to 3% by weight
	antiredepositing agent	0 to 3% by weight
40	polymer	0 to 10% by weight
	complex-binding agent	0 to 10% by weight

45 The extent of applicability of the invention appears from the following detailed description. It should, however, be understood that the detailed description and the specific examples are merely included to illustrate the preferred embodiments, and that various alterations and modifications within the scope of protection will be obvious to persons skilled in the art on the basis of the detailed description.

50 Detailed Description of the Invention

55 The laundry detergent tablets according to the invention are primarily intended for use in ordinary household washing machines, including both washing machines of the toploader-type, where the laundry and the detergent are introduced in the top of the washing machines in a vertically arranged drum, and the frontloader-type, where the laundry and the detergent are introduced in a horizontally arranged drum through a front door.

Washing machines of the toploader-type are preferably used in Asia, Australia and USA, where low washing temperatures are traditionally used, often in the range of from 15 to 20°C, which presents particular requirements to the

disintegration period of the laundry detergent tablets.

Washing machines of the frontloader-type are especially used in Europe. As mentioned above, the trend is towards the use of a decreasing consumption of water combined with shorter and shorter washing periods as well as lower and lower temperatures (on the average 30 to 60°C compared to 60 to 90°C previously), which also presents high requirements to the disintegration period of the laundry detergent tablets.

The use of the laundry detergent tablets in the European front-loader-washing machines is encumbered with the problem that in certain types of machines the laundry detergent tablets may have a tendency to deposit in the front door with the result that they are not dissolved and therefore not completely effective. When used in such machines, it is therefore important that the laundry detergent tablets are dissolved quickly after being introduced into the washing drum.

Laundry detergent tablets according to the invention to be used in soft cold water, such as water of a hardness of 0 - 7°dH and a temperature of 15 - 20°C, are advantageously composed as follows:

nonionic tenside	0.1 to 6% by weight
amphoteric tenside	0.1 to 5% by weight
polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
layered silicate and/or alkali metal silicate, and/or zeolite	1 to 10% by weight
potassium carbonate	20 to 70% by weight
disintegrating agent	0.5 to 5% by weight
binder	1 to 5% by weight
enzyme	0.25 to 10% by weight
antiredepositing agent	0.25 to 3% by weight
antifoaming agent	0 to 1 % by weight
agent preventing the running of colour	0 to 3% by weight
complex-binding agent	0 to 10% by weight

Laundry detergent tablets according to the invention to be used in hard hot water, such as water of a hardness of 10 - 20°dH and a temperature of 30 - 60°C, are advantageously composed as follows:

nonionic tenside	0.1 to 10% by weight
amphoteric tenside	0.1 to 10% by weight

5	polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 40% by weight
10	layered silicate and/or alkali metal silicate, and/or zeolite	5 to 30% by weight
	potassium carbonate	15 to 40% by weight
	antiredepositing agent	0.25 to 3% by weight
15	polymer	1 to 10% by weight
	disintegrating agent	0.5 to 5% by weight
20	binder	1 to 8% by weight
	enzyme	0.25 to 10% by weight
25	antifoaming agent	0 to 1% by weight
	agent preventing the running of colour	0 to 3% by weight
30	sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate	0 to 10% by weight
35	complex-binding agent	0 to 10% by weight

40 The laundry detergent tablets according to the invention are based on a builder system of a polyfunctional carboxylic acid and/or a salt thereof, and layered silicate and/or alkali metal silicate and/or zeolite. The laundry detergent tablets are preferably zeolite-poor or zeolite-free, but if desired, they may contain a small amount of zeolite, such as 0.1 to 10% by weight, said zeolite beyond having a builder effect also having a liquid-absorbing effect and thereby being active in providing a free-flowing powder.

45 For a number of years, zeolite has on account of its good builder properties dominated the market as builder ingredient in compact detergents, but during recent years attempts have been made at reducing or avoiding the use thereof due to problems applying especially to cleaning plants due to accumulation of mud. Moreover high concentrations of zeolite may cause problems of discolorations of for instance dark textiles, where the white zeolite particles are clearly visible.

50 The laundry detergent tablets are based on a particular tenside system comprising both nonionic tenside and amphoteric tenside, preferably in a weight ratio of 0.1:1 - 10:1, such as 0.2:1 - 5:1, for instance 1:1 - 2:1. The use of this particular tenside system in combination with the zeolite-poor or -free builder system results surprisingly in a high washing effect combined with a good storage stability as well as a rapid solubility. As it appears from the following comparison test, the washing effect of the laundry detergent tablets according to the invention is fully abreast of and in some cases even superior to the washing effect of the best products in the market.

55 The nonionic tenside is advantageously included in an amount of from 2.5 - 10.0, such as from 3.5 - 8.0, for instance from 4.5 - 6.0% by weight, and may in principle be selected from all the conventional nonionic tensides usually produced by condensation of a hydrophilic alkylene oxide with a hydrophobic compound. Examples of alkylene oxides are ethylene oxide, propylene oxide and/or butylene oxide, and examples of hydrophobic compounds are alcohols, thioalcohols, dioles, fatty acids, fatty acid amides, alkane sulphonamides, alkylamines as well as alkylphenols.

Among the nonionic tensides, especially ethoxylated compounds of primary and secondary fatty alcohols are preferred, said compounds being selected from linear fatty alcohols, oxoalcohols and alcohols prepared by the Ziegler-process, i.e. fatty alcohols with an even number of carbon atoms. Particularly preferred are condensation products having 2 to 40 moles of ethylene oxide (EO), preferably 3 to 16 moles of EO per mole of fatty alcohol selected from linear fatty alcohols, such as for instance C₁₂₋₁₈coco alcohols, C₁₆₋₁₈tallow alcohols, oleyl alcohol or other native alcohols as well as mixtures thereof.

Examples of nonionic tensides are oleyl monoethanol amide + 4 EO and ethoxylated C₁₂-C₁₄ fatty alcohol + 7 EO as well as the lower C₈₋₁₂alkyl polyglycosides of the so-called APG-types which are of a vegetable origin. Such compounds are very friendly to the skin and furthermore environmentally acceptable as they are completely biodegradable.

Petrochemically based tensides may, however, also be used, said tensides being very inexpensive as well as available in many different types and variants.

The amphoteric tenside used is advantageously included in an amount of from 0.5 - 7.0, such as from 1.5 - 6.0, for instance from 2.5 - 5.0% by weight, and may in principle be selected from all conventional amphoteric tensides. Examples are betaine derivatives, imidazoline derivatives, alkyl polyamine carboxylates, alkyl iminodipropionates etc. Specific examples are for instance oleoamphopolycarboxy glycinate, lauramidopropyl betaine and coco amidopropyl betaine.

In addition to the nonionic tenside and the amphoteric tenside, it is, if desired, possible to include a small amount of an anionic tenside in the laundry detergent tablets according to the invention. As anionic tenside it is possible to use any anionic tenside conventionally included in laundry detergents and not having a disadvantageous influence on the laundry detergent tablets. Thus, a too high amount of anionic tenside may result in formation of insoluble compounds at higher degrees of the water hardness, which results in a reduced washing effect.

The polyfunctional carboxylic acid and/or the salt thereof used is advantageously included in an amount of from 20.0 - 40.0, such as from 21.0 - 37.0, for instance from 22.5 - 35.0% by weight. Polyfunctional carboxylic acids may as a general rule be defined as such acids which beyond the one obligatory carboxylic acid group also contain at least one further functional group selected from carboxyl and hydroxy. The polyfunctional acids may furthermore also contain nitrogen. Non-limiting examples of such polyfunctional carboxylic acids are citric acid, nitrilotriacetic acid (NTA), ethylene diamine tetraacetic acid (EDTA) and isoserine diacetic acid (ISDA), of which citric acid is preferred. Citric acid is available in form of trisodium citrate dihydrate, anhydrous trisodium citrate as well as anhydrous monosodium citrate. Trisodium citrate dihydrate is particularly preferred.

Layered silicate and/or alkali metal silicate and/or zeolite are furthermore included as obligatory ingredient in the laundry detergent tablets according to the invention. Layered silicate and/or alkali metal silicate and/or zeolite are advantageously included in an amount of from 2.0 - 25.0, such as from 5.0 - 22.5, for instance from 10.0 - 20.0% by weight. Layered silicate or a combination of layered silicate and a small amount of alkali metal silicate are preferably used.

An example of alkali metal silicate is sodium silicate, preferably sodium silicate of the composition Na₂O:SiO₂ in the ratio of 1:1 - 1:3.5, preferably 1:2 - 1:3.5. It is also possible to use mixtures of silicates having a varying content of alkali, such as for instance a mixture of Na₂O:SiO₂ = 1:2.5 and Na₂O:SiO₂ = 1:2.

An example of a commercial product is sodium disilicate with the composition Na₂O:SiO₂ = 1:2, which for instance is sold by Crossfield Chemie B.V., Eijsden, the Netherlands.

As zeolite (aluminosilicate) all known types of aluminosilicates may in principle be used. Examples of such types are mentioned in Danish printed accepted publication No. 151,231, Danish printed accepted publication No. 154,827 and EP Offenlegungsschrift No. 289,767.

Particularly preferred as zeolite component in the laundry detergent according to the invention is zeolite A of the formula Na₁₂(AlO₂)₁₂·(SiO₂)₁₂·xH₂O, in which x = 20-30, preferably x = 27.

The potassium carbonate used is advantageously included in an amount of from 10.0 - 50.0, such as from 15.0 - 40.0, for instance from 20.0 - 35.0% by weight.

In the laundry detergent tablets according to the invention, especially those to be used in hard and/or hot water, a small amount of sodium bicarbonate and/or potassium bicarbonate or alternatively sodium carbonate and/or potassium carbonate or a mixture thereof may advantageously be included, such as in an amount of from 2.5 - 10.0, for instance from 4.0 - 7.0% by weight.

Furthermore, one or more ingredients are advantageously included in the laundry detergent tablets according to the invention, said ingredients being selected from disintegrating agents, binders, enzymes, antifoaming agents, agents preventing the running of colour, antiredepositing agents, polymers and complex-binding agents. Such agents are completely conventional.

Disintegrating agents swell at contact with water thereby accelerating the disintegration of the tablets. Examples of disintegrating agents are starch derivatives, cellulose compounds, polyvinyl pyrrolidone compounds, polyvinyl polypyrrolidone compounds, bentonite compounds, alginates, gelatine and pectines. The amount of disintegrating

agent is typically in the range of 1.5 - 4.0, such as 1.8 - 3.2, for instance 2.0 - 3.0% by weight.

In order to improve the dissolving period additionally, it is furthermore possible to use a small amount of an organic carboxylic acid, preferably a polyfunctional organic carboxylic acid, such as for instance maleic acid and the hydroxy carboxylic acids malic acid, citric acid and tartaric acid, which together with the carbonate and/or bicarbonate used result in an effervescent effect when the tablets come into contact with water.

Examples of binders are polyethylene glycol, preferably of a molecular weight in the range of 200 - 10,000, glycerol, natural gums, for instance acacie and gacanth, as well as sugars, such as glucose and sucrose. These compounds are described in greater detail in "Die Tablette" by Dr. W.A. Ritschel, published by Cantor KG/Aulendorf, Württ, Germany. The amount of binder is typically in the range of 2.0 - 6.0, such as 3.0 - 5.0% by weight.

Examples of enzymes are mixtures of specifically acting types, such as proteases, carbohydrases, esterases, lipases, oxidoreductases, catalases, peroxidases, ureases, isomerases, lyases, transferases, desmolases or nucleases, which are described further inter alia in US-PS Nos. 3,519,570 and 3,533,139.

Commercial products of such enzymes are often available as aqueous solutions, powders or granulates together with diluents, such as sodium chloride, sodium sulphate or specific phosphates. Particularly preferred for use in the laundry detergent tablets according to the invention are dust-free granulates, such as the T-granulates sold by Novo Nordisk A/S, Bagsvaerd, Denmark.

Especially preferred among these T-granulates are proteolytic enzymes sold under the trade names ESPERASE®, SAVINASE® and ALCALASE®, amylolytic enzymes sold under the trade name TERMAMYL®, lipolytic enzymes sold under the trade name "LIPOLASE" and "LIPOLASE ULTRA" and cellulytic enzymes sold under the trade name CELLUZYME®, as well as mixtures thereof.

Further examples are the commercial product Durazym which is a proteolytic enzyme, and the commercial product Duramyl, which is an amylolytic enzyme. These products have very good storage stability and are also sold by Novo Nordisk A/S.

The total amount of enzymes is advantageously in the range of 0.5 - 8.0, such as 1.0 - 6.5% by weight.

Examples of antifoaming agents are both silicone-free and silicone-based compounds. Among the silicone-based compounds, organo polysiloxane compounds are preferred. An example of a silicone-based organo polysiloxane product is SP 30, which is a powder product sold by Wacker Chemie GmbH, Germany. Moreover, DC2-3485 can be mentioned which is a silicone compound sold by N.V. Dow Corning Europe S.A., Brussels, Belgium. When antifoaming agents are included, the amount thereof is advantageously in the range of 0.15 - 0.60, such as 0.20 - 0.55% by weight.

Examples of agents preventing the running of colour are polyvinyl pyrrolidone (PVP), for instance in form of the product SOKALAN® HP 50 sold by BASF AG, Ludwigshafen, Germany. When agents preventing the running of colour are included, the amount thereof is typically in the range of 0.01 - 1.5, such as 0.01 - 1.0, for instance 0.1 - 0.5% by weight.

Antiredepositing agents are advantageously included in the laundry detergent tablets according to the invention for preventing a redepositing of soil particles. Examples thereof are cellulose colloids, such as carboxy methyl cellulose and methyl hydroxy cellulose. The amount of antiredepositing agent is typically in the range of 0.5 - 2.5, such as 1.0 - 2.0% by weight.

Examples of polymers advantageously forming part of laundry detergent tablets for washing in hard and/or hot water are polyanionic polymers, such as the ones described in the US-PS Nos. 3,308,067, 3,723,322, 4,144,226 and 4,146,495 as well as GB-PS No. 1,596,756. Among these polymers, homo- and/or copolymeric carboxylic acids are preferred, as well as the sodium or potassium salts thereof. The amount of polymer used is typically in the range of 1.0 - 9.0, such as 3.0 - 7.0, for instance 4.0 - 6.0% by weight.

Non-limiting examples are polymerisates of acrylic acid, hydroxy acrylic acid, maleic acid, itaconic acid, mesaconic acid, acotinic acid, methylene malonic acid, citraconic acid and the like as well as copolymerisates of the above carboxylic acids together or copolymerisates thereof with other ethylenically unsaturated compounds, such as ethylene, propylene, isobutylene, vinyl alcohol, vinyl methylether, furan, acrolein, vinyl acetate, acrylamide, acrylonitrile, methacrylic acid and crotonic acid.

The molecular weight of the homo- and copolymers is in the range of about 1,000 - 1,000,000, preferably 2,000 - 250,000 and most preferred 5,000 - 100,000.

As polycarboxylate, especially the sodium salt of acrylic/maleic acid copolymer of a molecular weight of about 70,000 is preferred. SOKALAN® CP 5 sold by BASF AG, Ludwigshafen, Germany is an example of a commercial product of such a composition. In addition, the Norasol® products can be mentioned, which are sold by NorsoHaas, France, such as Norasol WL 2B, which is a co-granulate of about 30% polyacrylate of a molecular weight of 4,500 and bicarbonate.

Furthermore it is possible, if desired, to include complex-binding agents and precipitation inhibitors, such as EDTA (ethylene diamine tetraacetic acid), DETPA (diethylene triamine pentaacetic acid), HEEDTA (hydroxy ethylethylene diamine triacetic acid), ISDA (isoserine diacetic acid) and phosphonates, i.e. phosphonic acids or salts thereof. A particular example of a phosphonate is Dequest 2016 D, which is tetrasodium-1-hydroxyethylene-(1,1-diphosphonate)

with a content of active acid of about 60%, and which is sold by Monsanto S.A., Brussels, Belgium. Regarding a more detailed description of complex-binding agents, reference is made to DK-PS No. 167,363.

Other ingredients, which, if desired, may be included in the laundry detergent tablets according to the invention, are further conventional tablet adjuvants, optical brightener, bleaching agents, bleaching agent activators, agents making the textiles soil-repelling, antiagglutinants, rinsing aids, dyes, deodorising agents, antioxidants and perfume.

The laundry detergent tablets according to the invention are prepared in a completely conventional manner.

Thus, for instance a basic powder fraction is mixed, which contains a polyfunctional carboxylic acid and/or a salt thereof, layered silicate and/or alkali metal silicate and/or zeolite, potassium carbonate and optionally sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate, binder, agents preventing the running of colour, antiredepositing agent, complex-binding agent, and polymer as well as other optional pulverulent ingredients.

Moreover a fraction of liquid nonionic tenside is provided, whereby - in case the tenside exists in solid form - heating is performed to above the melting point, for instance to about 40 - 60°C, such as 45 - 55°C.

Finally, a residue fraction is mixed, said fraction containing an amphoteric, pulverulent tenside and optionally a disintegrating agent, an enzyme as well as other ingredients, such as an antifoaming agent which should be subjected to the least possible heat effect and load during the processing in order not to harm the stability of the components.

The tenside fraction is sprayed onto the basic powder fraction in a suitable mixer while it is continuously stirred for about 5 - 18 minutes depending on to the mixer used, whereafter the residue fraction is added while stirring for about additionally 2 - 10 minutes. As a result a free-flowing, slightly granulated powder is obtained of a bulk density of about 600 - 900 g/l.

The pulverulent mixture is compressed in a tableting machine, such as KORSCH EK III-type, into tablets of the desired weight and size.

The invention is explained in detail in the following examples.

Example 1

In the manner described above, laundry detergent tablets according to the invention were produced with the compositions CL1 - CL11 stated below in Table 1. The laundry detergent tablets were compressed on a tableting machine of the KORSCH EK III-type by means of a pressure of 50 - 60 kN. The tablets had a circular cross section of a diameter of 40 mm, a height of 12 - 14 mm and a weight of 25 g.

The tablet strength, the disintegration period as well as the increase in volume during storage were measured for each of the prepared laundry detergent tablets and are also indicated in Table 1 below.

The tablet strength was measured by way of a vertical downward penetration in the middle of the tablet at a speed of 50 mm/min by means of a flat 8 mm Ø steel piston on a Holland CT5-tablet strength testing apparatus, and both the initial tablet strength as well as the tablet strength after storage under accelerated aging conditions at 35°C and 75% RH for one week, two weeks and four weeks were measured.

During the curing of the laundry detergent tablets within the first 24 hours, a certain increase of the pressure is allowable, but thereafter the pressure should not increase substantially during storage because the dissolving period would thereby be disadvantageously increased.

The disintegration period was measured by placing the tablet in a wire basket of stainless steel and of a circular cross section of a diameter of 50 mm. The steel basket was placed in a 1000 ml beaker, into which 900 ml of 20°C thermostat water (11 °dH) was filled. Stirring was carried out at 1000 rpm. The tablets have disintegrated when tablet residues can no longer be observed in the wire basket. Both the initial disintegration period as well as the disintegration period after storage for one, two and four weeks under accelerated aging conditions at 35°C and 75% RH were measured. The disintegration period should not be considerably increased during storage.

Finally, the percentage increase in volume of the laundry detergent tablets was measured by means of a Mitutoyo-slide gauge after storage under accelerated aging conditions at 35°C and 75% RH for four weeks.

The percentage increase in volume of the laundry detergent tablets should be as small as possible, because a small percentage increase in volume indicates a good storage stability.

Table 1

Ingredient	CL 1 % by weight	CL 2 % by weight	CL 3 % by weight	CL 4 % by weight	CL 5 % by weight	CL 6 % by weight	CL 7 % by weight	CL 8 % by weight	CL 9 % by weight	CL 10 % by weight	CL 11 % by weight
Citrate ADM-fine	35.00	35.00	35.00		25.00	25.00	35.00	35.00	25.00	22.50	35.00
Citrate gadot-fine				35.00							
K ₂ CO ₃ , NDH, gran. (coarse)	22.90	25.40	10.00	25.15							22.90
K ₂ CO ₃ , SCPA (fine)	20.00	20.00	35.40	23.00	25.50	31.45	20.00	20.00	23.50	26.95	15.00
NaHCO ₃											
KHCO ₃							6.45				
SKS-6 flower	2.00	2.00	2.00								
SKS-powder				2.00		20.00	10.00	10.00	25.00	22.50	
SKS-6 gran.					25.00						
Sokalan CP7					5.00						
Sokalan CP5						5.00	5.00		5.00	5.00	
Noradol WL 2B								14.70			
Avicel	3.00	3.00	3.00		1.00		3.00	3.00	3.00	3.00	3.00

Ingredient	CL 1 % by weight	CL 2 % by weight	CL 3 % by weight	CL 4 % by weight	CL 5 % by weight	CL 6 % by weight	CL 7 % by weight	CL 8 % by weight	CL 9 % by weight	CL 10 % by weight	CL 11 % by weight
Kollidon						2.00					
Pluriol F 1500	3.00		3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	5.00
Soap HB 3										1.00	
PEG 6000		3.00									
Amfolak X07-SD80	2.50	2.50	2.50	2.50	5.00	5.00	5.00	5.00	5.00	4.00	2.50
OMA-4	4.50	4.50	4.50	4.50	6.00	6.00	6.00		6.00	6.00	4.50
Wessalith CD											5.00
Imbentin AG124s/060								6.00			
Glycerin 85.5%	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
Tinopal CBSX	0.10	0.10	0.10	0.10							0.10
Alcalase	2.00	1.00	1.00	1.00	1.50	1.50		1.00	1.50		2.00
Termamyl	1.00										1.00
Lipolase									2.00	1.50	3.00
Lipolase Ultra	3.00						1.50				

Ingredient	CL 1 % by weight	CL 2 % by weight	CL 3 % by weight	CL 4 % by weight	CL 5 % by weight	CL 6 % by weight	CL 7 % by weight	CL 8 % by weight	CL 9 % by weight	CL 10 % by weight	CL 11 % by weight
Savinase 6.0 Tx							1.50			1.50	
CMC		2.50	2.50	2.50			2.00	1.00		2.00	
DC2-3485				0.25				0.25			
Antifoaming agent SP30							0.50				
Perfume						0.05	0.05	0.05		0.05	
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Pressure (measured in N) start	94	71	42	64	86	71	64	74	74	61	72
Pressure 1 week	124	91	69	87	110	84	83	122	96	66	67
Pressure 2 weeks	166	98	89	106	105	98	81	118	80	48	83
Pressure 4 weeks	210	114	105	105	117	100	63	76	151	68	80
Diss. period (in min/sec) start	0.56	2.05	0.33	1.24	1.55	1.12	2.03	3.09	1.22	1.33	1.00

Ingredient	CL 1 % by weight	CL 2 % by weight	CL 3 % by weight	CL 4 % by weight	CL 5 % by weight	CL 6 % by weight	CL 7 % by weight	CL 8 % by weight	CL 9 % by weight	CL 10 % by weight	CL 11 % by weight
Diss. period 1 week	2.06	1.42	0.48	1.17	2.54	2.19	1.45	3.38	3.16	2.37	0.47
Diss. period 2 weeks	1.05	4.05	0.31	0.41	2.61	2.30	1.58	5.41	3.19	2.00	0.44
Diss. period 4 weeks	3.19	3.36	1.08	1.36	3.39	2.01	2.12	3.33	4.12	4.09	2.05
Volt 4 weeks	5%	9%	3%	4%	8%	7%	6%	8%	6%	8%	9%

Footnotes to Table 1:

Citrate ADM-fine:

trisodium citrate dihydrate, fine granulate, sold by Archer Daniels Midland Company, Kent, United Kingdom

Citrate gadot-fine:

trisodium citrate dihydrate, fine granulate, sold by Gadot, Israel

K₂CO₃-NDH, granulate (coarse):

potassium carbonate, 99,100 granulate sold by Des Proanits Chemique, Harbonnieres, France

K₂CO₃-SCPA (fine):

potassium carbonate, fine granulate sold by SCPA, Paris Cedex, France

SKS-6 flour:

layered silicate in form of flour, ground SKS-6, sold by Hoechst AG, Frankfurt am Main, Germany

SKS-6 powder:

pulverulent layered silicate sold by Hoechst AG, Frankfurt am Main, Germany

SKS-6 granulate:

granulated layered silicate sold by Hoechst AG, Frankfurt am Main, Germany

Soap HB 3:

soap granulate HB3 based on vegetable oils and sold by Hirtler GmbH, Heitersheim, Germany

PEG 6000:

polyethylene of a molecular weight of 6,000

Amfolak X07-SD80:

oleo amphopolycarboxy glycinate, 80% active ingredient sold by Akzo Nobel Surface Chemistry AB, Stenungsund, Sweden

OMA-4:

oleyl monoethanolamide + 4 EO sold by Akzo Nobel Surface Chemistry AB, Stenungsund, Sweden

Wessalith CD:

71.0% zeolite A, 20.5% water, 5.4% polycarboxylate and 2.4% nonionic tenside (ethoxylated fatty alcohol), sold by Degussa AG, Frankfurt, Germany

Imbentin AG124s/060:

ethoxylated lauryl-myristyl alcohol sold by Dr. W. Kolb AG, Hedingen, Switzerland

Tinopal CBSX:

optical brightener sold by Ciba-Geigy, Basel, Switzerland

Alcalase:

proteolytic enzyme sold by Novo Nordisk A/S, Bagsvaerd, Denmark

Sokalan CP7:

acrylic acid/maleic acid-copolymerisate in form of the sodium salt of a molecular weight of about 50,000 sold by BASF AG, Ludwigshafen, Germany

Sokalan CP5:

acrylic acid/maleic acid-copolymerisate in form of the sodium salt of a molecular weight of about 70,000 sold by BASF AG, Ludwigshafen, Germany

Norasol WL 2B:

co-granulate of 30% active LMW 45 N - polyacrylate of a molecular weight of 4.500 and bicarbonate sold by NorsoHaas S.A., Verneuil-en-Halatte, France

Avicel PH 200:

microcrystalline cellulose sold by FMC Corp., Philadelphia, USA

Kollidon:

polyvinyl pyrrolidone, 80% active ingredient sold by BASF AG, Ludwigshafen, Germany

Pluriol E 1500:

polyethylene glycol of a molecular weight of 1.500 sold by BASF AG, Ludwigshafen, Germany

Termamyl:

amylolytic enzyme sold by Novo Nordisk A/S, Bagsvaerd, Denmark

Lipolase:

lipolytic enzyme sold by Novo Nordisk A/S, Bagsvaerd, Denmark

Lipolase Ultra:

lipolytic enzyme sold by Novo Nordisk A/S, Bagsvaerd, Denmark

Savinase 6.0 Tx:

proteolytic enzyme sold by Novo Nordisk A/S, Bagsvaerd, Denmark

CMC:

carboxymethyl cellulose

DC2-3485:

silicone-based, pulverulent antifoaming agent sold by N.V. Dow Corning Europe S.A., Brussels, Belgium

Antifoaming agent SP30

silicone-based organopolysiloxane sold by Wacker Chemie GmbH, Germany

Example 2

The present example illustrates a test carried out at an independent institute, CTTN-IREN INSTITUTE, Ecully Cedex, France, for comparing the washing effect between the present laundry detergent tablets and one of the best products in the market, Persil Megaperls Color supplied by Henkel. The latter product is available in form of pearls and has a zeolite content of more than 30%, a content of anionic tensides of from 5 to 15%, a content of nonionic tensides of from 5 to 15% and a content of polycarboxylate and soap of less than 5% (according to the label on the packing).

The following test conditions were employed:

Washing machine	FOM 71 Wascator
Programme	Standard (without prewash)
Temperature	40°C and 60°C
Water hardness	28°F (= 15.7°dH) (Ca:Mg = 4:1)
Amount of fabric	3.5 kg normally soiled laundry
Amount of water	20 l in the main wash
Number of washings	3, i.e. the numerical values are the average of 24 measurements where each test swatch was measured on 4 x 2 areas.

The results appear from the following Table 2. The reflection values indicated in the table indicate the efficiency of the laundry detergents used for removing the impregnated stains. A high value means a good efficiency, the theoretically highest possible value being 100 corresponding to a completely white surface.

Moreover, the degree of brightness was spectrophotometrically measured at repeated washings of a swatch of white fabric. The higher the measured value is, the higher the degree of brightness.

Table 2

Type of soil	Code for test swatch	Persil Mega-perls 80 g pearls 40°C	CL 9 2x25 g tablets 40°C	Persil Mega-perls 80 g pearls 60°C	CL 9 2x25 g tablets 60°C
Pigment stains on cotton	Test Fabric*	32.7	37.5	55.6	55.9
	WFK 10 C	32.0	34.4	72.0	76.5
	IEC 106	28.1	28.6	41.1	41.2
	EMPA 101	22.4	25.8	35.3	32.8
	Average	28.8	31.6	51.0	51.6
Pigment stains on mixed fibres	Test Fabric**	41.0	30.0	59.8	41.1
	WFK 20 C	22.9	29.8	25.1	39.1
	EMPA 104	22.1	27.6	34.2	39.3
	Average	28.7	29.1	39.7	39.8
Protein stains	EMPA 111	65.3	94.1	89.1	99.6
	EMPA 112	38.3	47.5	52.9	52.5
	EMPA 116	49.8	58.6	63.4	67.8
	Average	51.1	66.7	68.5	73.3
Bleachable stains	CTTN-TYPE	0.0	0.0	0.0	0.0
	EMPA 222	4.7	2.4	16.5	11.6
	EMPA 114	20.4	9.0	24.1	9.8
	Average	8.4	3.8	13.5	7.1
Degree of brightness	EMPA 221	5.7	5.5	10.4	10.3

Footnotes to Table:

Test swatch	Type of soil
Test Fabric*	Vegetable and mineral oil, starch, carbon black, oleic acid, morpholine on cotton
WFK 10 C	Standard soil on cotton
IEC 106	Mineral oil and carbon black on cotton
EMPA 101	Soot/vegetable oil on cotton
Test Fabric**	Vegetable and mineral oil, starch, carbon black, oleic acid, morpholine on mixed fibres
WFK 20 C	Standard soil on mixed fibres
EMPA 104	Olive oil and carbon black on mixed fibres
EMPA 111	Blood
EMPA 112	Cocoa
EMPA 116	Blood, milk and cocoa on cotton
CTTN-type	Tea
EMPA 222	Untreated (non-bleached) cotton
EMPA 114	Wine
EMPA 221	White cotton without soil

As it appears from the results, the laundry detergent tablets according to the invention show a washing effect which is completely abreast of and even slightly better than Persil Megaperls Color. This is even more surprising as a considerably smaller amount of tenside is included in the laundry detergent tablets according to the invention compared to Persil Megaperls Color. Thus a total of 5.0 g of tenside is included in the amount of CL9 used, whereas a total of 8 to 24 g of tenside is included in the amount of Persil Megaperls Color used (according to the label on the packing). The degree of brightness is almost the same for both products, and a tendency towards a higher degree of brightness applies when increasing the washing temperature.

Example 3

The present example illustrates a test carried out for a comparison of the washing effect between the laundry detergent tablets according to the invention and two of the best products in the market Persil Megaperls Color sold by Henkel and Ariel Futur Color sold by Procter & Gamble. Ariel Futur Color is a pulverulent product and contains 5 to 15% of anionic tensides, 5 to 15% of nonionic tensides, 5 to 15% polycarboxylates, 15 to 30% zeolites and less than 5% phosphonates (according to the label on the packing).

The following test conditions were employed:

Washing machine:	Bauknecht Öko-matic WA 1200
Programme:	Standard (without prewash)
Temperature:	40°C

	Water hardness:	25°dH
	Amount of fabric:	3.5 kg clean terry towels
5	Test swatches:	Pre-impregnated pieces of fabric, 10 x 10 cm, supplied by WFK Krefeld, Germany
10	Number of washings:	6, i.e. the numerical values indicated in the Table are an average of 54 measurements, each test swatch being measured on 3 x 3 areas
15	Spectrophotometer for measuring reflection values:	Minolta CR 200

20 The results appear from the following Table 3. The reflection values indicated in the table indicate the efficiency of the laundry detergents used for removing the impregnated stains. A high value means a good efficiency, the theoretically highest possible value being 100 corresponding to a completely white surface.

Table 3

Type of soil	Before washing	Persil Megaperls Color 84 g	Ariel Futur Color 80 g	CL 9 2x25 g tablets	CL 10 2 x 25 g tablets
Standard soil on cotton	39.93	75.86	78.22	74.74	76.16
Standard soil on mixed fibres	50.30	69.42	71.22	70.36	70.90
Sebum on cotton	40.22	75.18	77.23	72.61	73.64
Blood, milk, ink on cotton	12.71	51.46	52.66	48.26	52.07
Blood on cotton	19.40	78.40	69.44	72.48	76.80
Cocoa on cotton	41.38	65.69	64.08	58.74	59.76
Cocoa on mixed fibres	48.34	76.72	74.35	67.60	68.92
Pigment/oil	51.50	73.17	74.78	70.17	71.11
Grass on cotton	43.35	62.59	62.81	56.70	61.38
Average	38.57	69.83	69.42	65.74	67.86

As seen from the results, the effect of the laundry detergent tablets according to the invention is almost abreast of the known products for the tested types of soil. This is a surprisingly good result, as a considerable lower amount of tenside is used in the laundry detergent tablets according to the invention compared to the known products. Thus, 5.0 g of tenside is included in CL9 and 5.1 g of tenside in CL10, whereas Ariel Futur Color includes a total of from 8 to 24 g and Persil Megaperls Color of from 8.4 to 25.2 g (according to the labels on the respective packings).

Example 4

The present example as well as the following examples 5 and 6 illustrate tests carried out for a comparison of the washing effect of the laundry detergent tablets according to the invention and the detergent known from EP No. 0 482 627 (KAO).

The tests used the following formulations, the recipes being presented as powders as it was difficult to produce tablets on the basis of the recipes indicated in EP No. 0 482 627.

Recipes used:

Ingredient	KAO 1.1 % by weight	CL 8 % by weight
Citrate ADM-fine:		35.00
Wessalith CD	28.20	
K ₂ CO ₃ , SCPA (fine)	19.00	20.00
Aerosil	10.00	
Soda	1.00	
SKS-powder		10.00
Norasol LMW45	0.48	
Norasol WL2B 2B:		14.70
Avicel		3.00
Pluriol E 1500		3.00
PEG 6000	1.00	
Sodium sulphate, heavy	19.99	
Amfolak X07-SD80		5.00
Berol 173		
Lutensol A7N	19.33	
Imbentin AG124s/060		6.00
Glycerin 85.5%		1.00
Alcalase	1.00	1.00
CMC		1.00
DC2-3485 (anti-foaming agent)		0.25
Perfume		0.05
Total	100.00	100.00

Footnotes:

Aerosil:

Amorphous silicon oxide sold by Degussa AG, Frankfurt, Germany

Norasol LMW45:

Polyacrylate sold by NorsoHaas S.A., Verneuil-en-Halatte, France

Sodium sulphate, heavy:

Sodium sulphate

Lutensol A7N:

Highly ethoxylated linear fatty alcohol sold by BASF AG, Ludwigshafen, Germany

The following test conditions were employed:

Washing machine	Toploader, Goldstar 7.2 kg WF-1505AHP
Programme	Main wash 12 min.
Temperature	20°C
Water hardness	10°dH
Amount of fabric	1.5 kg normally soiled laundry
Number of washings	3, i.e. each numerical value indicated is an average of 27 measurements, each test swatch being measured on 3 x 3 areas.
Spectrophotometer for measuring reflection values	Minolta CR 200

The results appear from the following Table 4.

Table 4

Type of soil	Before washing	KAO 1.1 50 g powder	CL 8 50 g powder
Standard soil on cotton	38.50	55.89	54.18
Standard soil on mixed fibres	50.66	70.20	66.81
Sebum on cotton	38.31	61.57	57.85
Blood, milk, ink on cotton	12.80	35.38	37.00
Blood on cotton	21.64	66.36	62.22
Cocoa on mixed fibres	51.88	70.60	68.21
Pigment/oil	50.51	68.18	66.82
Oil/Pigment/Milk	50.84	65.63	64.98
Average	39.39	61.73	59.76

As illustrated in the above Table, the detergent according to EP No. 0 482 627 (KAO 1.1) showed a slightly better washing effect in connection with the tested types of soil compared to the laundry detergent tablets according to the invention. In this connection it should be mentioned that a total amount of tenside of 9.7 g is included in the KAO 1.1, whereas only a total of 5.0 g of tenside is included in CL8.

Example 5

Using the same procedure as disclosed in example 4 above, yet another comparison test was carried out, whereby, however, the following test conditions applied:

Washing machine	Bauknecht Öko-matic WA 1200
Temperature	40°C
Programme	Standard (without prewash)
Water hardness	18°dH
Amount of fabric	2 kg of normally soiled laundry + 1.5 kg of clean terry towels.
Number of washings	3, i.e. each numerical value indicated is an average of 27 measurements, each test swatch being measured on 3 x 3 areas.
Spectrophotometer for measuring reflection values	Minolta CR 200

The results appear from the following Table 5.

Table 5

Type of soil	Before washing	KAO 1.1 50 g powder	CL 8 2 x 25 g tablets
Standard soil on cotton	38.50	57.56	51.70

Table 5 (continued)

Type of soil	Before washing	KAO 1.1 50 g powder	CL 8 2 x 25 g tablets
Standard soil on mixed fibres	50.66	66.06	64.73
Sebum on cotton	38.31	73.46	62.10
Blood, milk, ink on cotton	12.80	43.71	47.77
Blood on cotton	21.64	67.82	72.03
Cocoa on mixed fibres	51.88	68.77	63.52
Pigment/oil	50.51	67.01	65.84
Oil/Pigment/Milk	50.84	79.75	78.23
Average	39.39	65.52	63.24

As illustrated in the above Table, the detergent according to EP No. 0 482 627 (KAO 1.1) showed a slightly better washing effect in connection with the tested types of soil compared to the laundry detergent tablets according to the invention. In this connection it should be mentioned that a total amount of tenside of 9.7 g is included in KAO 1.1, whereas only a total of 5.0 g of tenside is included in CL8.

Example 6

Like above yet another comparison test was carried out, whereby, however, the following test conditions applied, the washing, however, being carried out at 60°C.

Washing machine:	Bauknecht Öko-matic WA 1200
Temperature:	60°C
Programme:	Standard (without prewash)
Water hardness:	18°dH
Amount of fabric:	2 kg of normally soiled laundry + 1.5 kg of clean terry towels.
Number of washings:	3, i.e. each numerical value indicated is an average of 27 measurements, each test swatch being measured on 3 x 3 areas.
Spectrophotometer for measuring reflection values:	Minolta CR 200

The results appear from the following Table 6.

Table 6

Type of soil	Before washing	KAO 1.1 50 g powder	CL 8 50 g powder
Standard soil on cotton	38.50	60.78	62.35
Standard soil on mixed fibres	50.66	68.16	65.77
Sebum on cotton	38.31	75.75	63.74
Blood, milk, ink on cotton	12.80	47.88	53.68
Blood on cotton	21.64	71.13	77.96
Cocoa on mixed fibres	51.88	69.58	64.98
Pigment/oil	50.51	69.69	70.54
Oil/Pigment/Milk	50.84	80.99	82.30
Average	39.39	67.99	67.67

As shown in the above Table, the detergent according to EP No. 0 482 627 (KAO 1.1) displayed a washing effect in connection with the tested types of soil comparable with the laundry detergent tablets according to the invention. This is a surprisingly good result, as the total amount of tenside in KAO 1.1 is considerably higher than the total amount

of tenside in the inventive laundry detergent tablets.

Example 7

The present example illustrates a test carried out for a comparison of the dissolving period for the laundry detergent tablets according to the invention (CL 8) with the detergent according to EP No. 0 482 627 (KAO 1.1).

The test used 2x25 g of CL 8-tablets as well as 2 x 25 g KAO 1.1-tablets.

The dissolving period was measured at a washing in a washing machine of the type Bauknecht Öko-matic WA 1200 using a standard washing programme at 40°C and an amount of water of about 10 l in the main wash. The tablets were at the dissolving test placed in a wide-meshed washing bag of 18 x 10 cm in order to prevent the tablets from depositing in the door of the washing machine. The washing was carried out on 3.5 kg clean terry towels.

The results appear from the following Table 7.

Table 7

Dissolving period - percentage residue amount
of tablet over time

Time (min)	CL 8	KAO 1.1
0	100%	100%
2	90%	110%
4	54%	99%
6	12%	96%
8	0%	89%
10	0%	82%
12	0%	68%
14	0%	62%
16	0%	57%
18	0%	50%
20	0%	44%
22	0%	40%
24	0%	37%
26	0%	26%
28	0%	23%

As it appears from the test which was carried out two hours after the preparation of the tablets, it took the KAO 1.1-tablets a very long time to dissolve. At the beginning of the first rinsing cycle after 28 minutes, a residue amount of 23% thus remained, which corresponds to a tablet weight of 10.8 g. Unlike the known products, the laundry detergent tablets according to the invention had dissolved completely after 8 minutes.

The above description of the invention makes it obvious that it can be varied in many ways. Such variations are

not to be considered a deviation from the scope of the invention, and all such modifications which are obvious to persons skilled in the art are also to be considered comprised by the scope of the succeeding claims.

Claims

1. Laundry detergent tablets, **characterized** in that in addition to optional, usual adjuvants and additives they comprise

nonionic tenside	0.1 to 15% by weight
amphoteric tenside	0.1 to 15% by weight
polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
layered silicate and/or alkali metal silicate and/or zeolite	1 to 30% by weight
potassium carbonate	5 to 70% by weight

2. Laundry detergent tablets as claimed in claim 1, **characterized** in that in addition to optional, usual adjuvants and additives they comprise one or more ingredients selected from sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate, disintegrating agents, binders, enzymes, antifoaming agents, agents preventing the running of colour, antiredepositing agents, polymers and complex-binding agents.

3. Laundry detergent tablets as claimed in claim 1, **characterized** in that in addition to optional, usual adjuvants and additives they comprise

nonionic tenside	0.1 to 15% by weight
amphoteric tenside	0.1 to 15% by weight

5	polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
	layered silicate and/or alkali metal silicate and/or zeolite	1 to 30% by weight
10	potassium carbonate	5 to 70% by weight
	disintegrating agent	0.5 to 5% by weight
15	binder	1 to 8% by weight
20	sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate	0 to 10% by weight
	enzyme	0 to 10% by weight
25	antifoaming agent	0 to 1% by weight
	agent preventing the running of colour	0 to 3% by weight
30	antiredepositing agent	0 to 3% by weight
	polymer	0 to 10% by weight
	complex-binding agent	0 to 10% by weight
35	4. Laundry detergent tablets as claimed in claim 1. characterized in that in addition to optional. usual adjuvants and additives they comprise	
40	nonionic tenside	0.1 to 6% by weight
	amphoteric tenside	0.1 to 5% by weight
45	polyfunctional carboxylic acid and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 50% by weight
50		
55		

layered silicate and/or alkali metal silicate and/or zeolite	1 to 10% by weight
potassium carbonate	20 to 70% by weight
disintegrating agent	0.5 to 5% by weight
binder	1 to 5% by weight
enzyme	0.25 to 10% by weight
antiredepositing agent	0.25 to 3% by weight
antifoaming agent	0 to 1% by weight
agent preventing the running of colour	0 to 3% by weight
complex-binding agent	0 to 10% by weight

5. Laundry detergent tablets as claimed in claim 1, **characterized** in that in addition to optional, usual adjuvants and additives they comprise

nonionic tenside	0.1 to 10% by weight
amphoteric tenside	0.1 to 10% by weight
polyfunctional carboxylic acid and/or a salt thereof calculated as tri- sodium citrate dihydrate	20 to 40% by weight
layered silicate and/or alkali metal silicate and/or zeolite	5 to 30% by weight
potassium carbonate	15 to 40% by weight
disintegrating agent	0.5 to 5% by weight
binder	1 to 8% by weight

	enzyme	0.25 to 10% by weight
5	antiredepositing agent	0.25 to 3% by weight
	polymer	1 to 10% by weight
10	sodium carbonate/bicarbonate and/or potassium carbonate/ bicarbonate	0 to 10% by weight
15	antifoaming agent	0 to 1% by weight
	agent preventing the running of colour	0 to 3% by weight
20	complex-binding agent	0 to 10% by weight

6. Laundry detergent tablets as claimed in claim 1, **characterized** in that in addition to optional, usual adjuvants and additives they comprise

25		
	nonionic tenside	2.5 to 8% by weight
30	amphoteric tenside	0.5 to 7% by weight
35	polyfunctional carboxylic acid, and/or a salt thereof calculated as trisodium citrate dihydrate	20 to 40% by weight
40	layered silicate and/or alkali metal silicate and/or zeolite	2 to 25% by weight
	potassium carbonate	10 to 50% by weight
45	antiredepositing agent	0.5 to 2.5% by weight
	disintegrating agent	1.5 to 4% by weight
50	binder	2 to 6% by weight
55		

	enzyme	0.5 to 7% by weight
5	antifoaming agent	0 to 0.8% by weight
	agent preventing the running of colour	0 to 1.5% by weight
10	sodium carbonate/bicarbonate and/or potassium carbonate/ bicarbonate	0 to 9% by weight
15	polymer	0 to 9% by weight
	complex-binding agent	0 to 10% by weight
20	7. Laundry detergent tablets as claimed in claim 1, characterized in that in addition to optional, usual adjuvants and additives they comprise	
25	nonionic tenside	4.5 to 6.0% by weight
	amphoteric tenside	2.5 to 5.0% by weight
30	citrate calculated as trisodium citrate dihydrate	22.5 to 35.0% by weight
35	layered silicate and/or alkali metal silicate	10.0 to 20.0% by weight
	potassium carbonate	20.0 to 32.0% by weight
40	disintegrating agent	2.0 to 3.0% by weight
	binder	3.0 to 5.0% by weight
45	enzyme	1.0 to 6.5% by weight
	cellulose colloid	1.0 to 2.0% by weight
50	polymer	4.0 to 6.0% by weight

	complex-binding agent	0 to 10% by weight
5	antifoaming agent	0 to 0.55% by weight
	agent preventing the running of colour	0 to 0.5% by weight
10	sodium carbonate/bicarbonate and/or potassium carbonate/bicarbonate	0 to 7.0% by weight

- 15 8. Laundry detergent tablets as claimed in any of the preceding claims 1 to 7, **characterized** in that the nonionic tenside is selected from condensation products of a hydrophilic alkylene oxide with a fatty alcohol, a fatty thioalcohol, a fatty acid, a fatty acid amide, an alkane sulphonamide, an alkylamine or an alkylphenole and alkylpolyglycosides.
- 20 9. Laundry detergent tablets as claimed in any of the preceding claims 1 to 8, **characterized** in that the amphoteric tenside is selected from oleoamphopolycarboxy glycinate, lauramidopropyl betaine and cocoamidopropyl betaine.

(19)



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(54) **Laundry detergent tablets**

(57) Laundry detergent tablets which in addition to usual adjuvants and additives comprise 0.1 to 15% by weight of nonionic tenside, 0.1 to 15% by weight of amphoteric tenside, 20 to 50% by weight of a polyfunctional carboxylic acid and/or a salt thereof, calculated as trisodium citrate dihydrate, 1 to 30% by weight of layered silicate and/or alkalimetal silicate and/or zeolite as well as 5 to 70% by weight of potassium carbonate. Further ingredients advantageously included in the laundry de-

tergent tablets are sodium carbonate and/or potassium carbonate, sodium bicarbonate and/or potassium carbonate, disintegrating agents, binders, enzymes, anti-foaming agents, agents preventing the running of colour, antiredepositing agents, polymers and complex-binding agents. The laundry detergent tablets have a good washing effect at the same time as they have a good storage stability and dissolve quickly in the washing water.

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EUROPEAN SEARCH REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	WO 95 20030 A (UNILEVER) 27 July 1995 * page 12, last paragraph - page 13, paragraph 1: claims 1-9 *	1-2	C11D1/94 C11D3/10 C11D3/12 C11D3/20 C11D17/00 C11D3/08
D.A	EP 0 482 627 A (KAO CORP.) 29 April 1992 * claims 1,5: tables 1,2 *	1-7	
P.A	EP 0 737 738 A (CLEANTABS) 16 October 1996 * page 4, line 26-36: claims 1,6,10 *	1-7	
A	EP 0 504 091 A (VIKING INDUSTRIES) 16 September 1992 * claims 1-3,9 *	1-7	
A	DATABASE WPI Week 9241 Derwent Publications Ltd., London, GB: AN 92-335478 XP002078351 & JP 04 239100 A (LION CORP.) , 26 August 1992 * abstract *	1-7	
D	& JP 04 239100 A (...)		
A	DATABASE WPI Week 9420 Derwent Publications Ltd., London, GB: AN 94-164283 XP002078352 & JP 06 108099 A (LION CORP.) , 19 April 1994 * abstract *	1-7	
D	& JP 06 108099 A (...)		
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 22 September 1998	Examiner Van Bellingen, I
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background C: non-written disclosure P: intermediate document</p> <p>I: theory or principle underlying the invention E: earlier patent document, but published on or after the filing date D: document cited in the application L: document cited for other reasons S: member of the same patent family, corresponding document</p>			

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